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MECHANICAL PROPERTIES OF PYROLYTIC GRAPHITE

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MECHANICAL PROPERTIES OF PYROLYTIC GRAPHITE

By

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GENERAL ELECTRIC

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

Although pyrolytic graphite has become widely known[1,6] as a material of unusual properties and great promise in aerospace technology, little appears to have been published recently recently regarding its mechanical properties. Aside from the data obtained during the early phases of its commercial development, much of the test data appear only in individual rather restricted test programs, and are not widely circulated[7,8].

Inasmuch as considerable refinement of the manufacturing process has occurred since the early test data were gathered there is some question as to how realistically these early data represent the properties of pyrolytic graphite as it is currently manufactured. Therefore, it became important to assess the mechanical properties of current production material using techniques appropriate for this brittle anistropic material.

II. PYROLYTIC GRAPHITE

It is important to consider in the beginning, the features of pyrolytic graphite which make it unusual and which make testing somewhat more difficult than conventional materials.

Pyrolytic graphite is a polycrystalline form of graphite deposited at high temperatures (ca 4000°F) by thermal decomposition of a simple hydrocarbon such as methane. The deposits consist of layers of wavy and kinked planes of hexagonally arranged carbon atoms, mutually parallel but randomly rotated about an axis perpendicular to the plane of the deposit. In customary terminology, this axis is known as the "c" axis or direction while the direction parallel to the planes is referred to as the a-b direction. The microstructures of the deposits vary with processing conditions and have intimate influence on the properties of the deposits[3]. In general, two representative classes of structures have become known. These are the so-called surface nucleated and continuously nucleated or regenerative pyrolytic graphite. Figure 1 illustrates the gross differences between them. Figure 1a shows the former type in which the so-called growth structure originates at the first deposited layer and is propagated in uninterrupted fashion to the top of the deposit. The regenerative structure however is continuously interrupted by additional nuclei or growth origins laid down throughout manufacture and presents the aspect shown in Figure 1b. It should be emphasized that all variations between these extremes can and are produced with associated effects on the material properties. Thus,

pyrolytic graphite is essentially a class of materials, the specific properties of each member being dependent on its characteristic microstructure.

The layered structure, with strong covalent bonding in the planes and weak electrostatic (van der Waals) bonding between the planes, leads to a high degree of anisotropy in all properties[4]. Because of its brittleness, pyrolytic graphite is subject to serious damage and premature failure if the surfaces of test specimens are not carefully finished. This further emphasizes the problems associated with alignment in mechanical tests.

In addition to the difficulties encountered in testing because of the inherent anisotropy and brittleness of pyrolytic graphite, process variations can also induce among other things, isolated nodules which serve as stress centers, high grain boundary angles and delaminations. All of these serve to lower the test values and to increase their scatter.

Proper selection of material, careful attention to machining and alignment and examination of fracture surfaces can serve however to reduce the scatter, to explain unexpectedly low values, and generally, to increase the working knowledge of pyrolytic graphite which is necessary to achieve the full potential of its mechanical properties.

With these considerations in mind a test program was developed to obtain reliable data on specimens characteristic of current production of the two general structural types described above. It was not considered in any sense a statistical evaluation, but was aimed instead of explaining the broad scatter band and relatively low values of room temperature measurement made earlier[7].

Previous experience[8] showed the necessity for selecting material not obviously defective, while other work[9] pointed out the need for careful machining and alignment of test specimens. Consequently, considerable attention was paid to details in these areas.

III. EXPERIMENTAL RESULTS AND DISCUSSION

The following measurements were carried out on both continuously nucleated and surface nucleated pyrolytic graphite as functions of temperature up to 5000°F: 1) ultimate tensile strength in the 'a' direction, 2) torsional strength in the 'a' direction, 3) flexure strength both parallel and perpendicular to the 'c' axis by three and four point loading, 4) elastic modulus in the 'a' direction and 5) linear thermal expansion in the 'a' and 'c' directions.

Material representative of current production was obtained from the Metallurgical Products Department, General Electric Co. (surface nucleated - SN) and the Raytheon Manufacturing Co., (regenerative or continuously nucleated - CN). Densities were 2.20 and 2.206 g/cc respectively measured by immersion in alcohol. Microstructures of the two types of material are shown in Figure 1a and 1b.

Test specimens were cut from flat plate raw stock and all surfaces were finished by grinding with a relatively soft wheel (SiC, 100 grit, H-bond, vitrified) rotating at 5000 surface ft/min.

Elevated temperatures were approached as rapidly as possible followed by a five minute soak at test temperature. In tests at 5000°F the apparatus was also heated as rapidly as possible, but the soak time included the period required to go from 4000°F to 5000°F. Thus all specimens tested at 5000°F were above the deposition temperature (4000°F) for about the same length of time.

A. ULTIMATE TENSILE STRENGTH

Ultimate tensile strength parallel to the planes was measured using an Instron testing machine equipped with a graphite resistance furnance capable of achieving 5000°F in a helium atmosphere. Viewing ports and telescopes permitted visual observation and measurement during testing. Tests at room temperature were run in laboratory atmosphere with transverse and longitudinal strain gages (Budd Co., #C61X1-M50A) attached using Eastman 910 cement. The tensile specimen was two inches wide at the gripping ends and narrowed to a gage section of 0.200" x 0.200". Overall length was six inches and effective gage length was 1.5465 inches. Cross head motion was 0.020 in/min. The procedure and specimen design were slightly modified at 5000°F by reducing the gage cross section to 0.200" deep by 0.125" wide and increasing the cross head speed to 0.050 in/min. This was done to insure gage length failure and to compensaute for the elongation of the specimen.

The results of the tensile tests are shown in Figure 2 and Table I. The values listed are all gage length failures except for the two so designated at 5000°F. Earlier values[7] are also shown at the 50 and 90% confidence levels for comparison.

B. TORSIONAL STRENGTH

Torsional strength is defined as the resistance of the a-b planes to rotation about the 'c' axis when torque is applied to the specimen in a direction parallel to the planes. Specimens were designed as shown in Figure 3 and subjected to torsional testing up to 5000°F. The faces of the

specimens fitted into similarly shaped wells in the ends of the graphite push rods of the Instron machine. No force was applied to the faces parallel to the 'c' axis of the specimen during testing. Cross head motion and thermal cycle were the same as for tensile tests. The results are listed in Figure 4 and Table II.

The higher values characteristic of the regenerative material reflect the increased 'interlocking' caused by the continued formation of new growth cones during deposition. Such interlocking reduces the anistropy of the material and is responsible for the differences in mechanical, electrical and thermal properties of surface nucleated and generative pyrolytic graphites.

C. FLEXURE STRENGTH

Flexure tests were carried out at temperatures up to 5000°F using both three and four point loading techniques with the last deposited surface of the material in tension. Tests were made with loading applied both parallel and perpendicular to the 'c' axis. Beams were 2-1/4" long and .200" square, all sides ground flat and parallel. In the three point loading tests, the span was 1-1/2", while in 4-point loading, the loading bars were 2 inches apart on the tensile side and 1 inch apart on the compressive side. Longitudinal and transverse strain gages were attached to several of the room temperature 4-point load tests to obtain tensile and compressive modulus data. Results are summarized in Tables I & II and Figures 5 & 6.

Differences between materials are most pronounced in the parallel orientation. Failure in the surface nucleated material was chiefly by

delamination near the neutral axis, the region of maximum shear stress. On the other hand, owing to its higher interlaminar shear strength, regenerative material failed by basal plane tension.

Some general conclusions can be drawn from the flexure test series: Four point load tests are likely to be lower than three point load tests for two reasons. First, somewhat higher shear stresses are associated with the former (between load and reaction pins) and, therefore, they tend to cause delaminations more readily (in the parallel orientation). Second, more of the specimen area is subject to the maximum tensile stress in the four point load test, a disadvantage in view of the critical nature of the surfaces in brittle materials. Because of its higher shear strength (see torsion data), the CN material is not as subject to delamination as the SN material. The differences in the flexure test results may thus be more indicative of differences in anisotropy rather than differences in tensile strength. This is also supported by the tensile test results.

A few observations can also be made relative to the variations noted within the flexure test series. First, beams tested in the parallel orientation are weaker than those tested in the perpendicular orientation. Second, beams tested by four point loading are weaker than those tested on three-point loading. Third, these differences are more pronounced in the SN than in the CN material. All of these phenomena are probably, to a substantial degree, a consequence of differences associated with the test configurations and differences in shear strengths of the two materials (see torsion test results).

In general, the use of flexure tests results as an index of tensile strength does not appear to be definitive. Factors such as resolved stresses, beam thickness, microstructure, method of loading and mode of failure all serve to confuse interpretation of results with respect to probable tensile strengths at failure. However, from a practical point of view, since flexural loading is common to many useful configurations, flexure test data must be obtained and interpreted with all of the above factors in mind. Thus, attractive as it appears to be from the standpoint of simplicity in performance and economy of material, the flexure test must be more carefully interpreted when used in connection with anisotropic materials such as are now under study in several vapor deposition programs. Microstructures, modes of failure and specimen dimensions among other things must be factored into the knowledge gained from such tests.

D. ELASTIC MODULUS

Elastic moduli were computed for both surface and continuously nucleated material from the strain gage data obtained at room temperature in both tension and four point bend tests, as well as from direct observation at elevated temperatures. These results are shown in Figure 7 and Table V. Here again a significant difference is seen as a result of microstructural variation. The surface nucleated material is somewhat stiffer throughout the temperature range than is the continuously nucleated material. On an absolute scale however, the difference is not large, both materials being characterized as having relatively low moduli, although they are from two to three times higher than a good grade of hot pressed graphite[10]. Strain

gage measurements on the four point bend specimens were used to calculated room temperature moduli for both tensile and compressive surfaces. These values are listed in Table V along with Poisson's ratios determined from dimensional changes occurring during room temperature tensile tests. The negative sign associated with these ratios is probably the result of flattening out of the wrinkles characteristic of the basal planes of pyrolytic graphite, the net result being an increase in the 'a' and 'b' direction dimensions and a decrease in the 'c' or interplanar dimension.

E. THERMAL EXPANSION

Results of thermal expansion measurements, made in both 'a' and 'c' directions for the two test materials are shown in Figures 8 and 9, respectively. As can be seen from the descending portion of the curve, there appears to be a permanent elongation for both grades of material, somewhat greater for the continuously nucleated material than for surface nucleated material. This is due to the annealing and flattening out of wrinkles in the basal planes which occurs. The effect of this on the 'c' direction expansion is to cause a partial reversal at elevated temperatures as the specimen is permitted to remain at test temperature. This is indicated by the downward turn at the end of the 'c' direction expansion curve. The calculated coefficients of expansion over the linear portions of the curves do not indicate very much effect of microstructure on the apparent thermal anisotropy ratios for the two materials. Certainly, anisotropy is a much less obvious factor than in the case of torsional and flexure tests.

IV. SURFACE PREPARATION AND TEST RESULTS

As is commonly observed in the case of brittle materials, machining and surface finishing procedures have a profound effect on test results. This is illustrated by the fact that tensile specimens, which failed at low stress values in room temperature tests, failed by basal plane failure at grips, an occurrence also noted in earlier test reports. Examination of such specimens after flame polishing[11] to bring out structural features, showed considerable delamination emanating from the edge of the grip hole (Figure 10). In effect, the specimen had been severely notched prior to test. Remachining of untested specimens, and retesting of broken specimens using fillet grips gave higher tensile strength values with failures occurring in the gage length. Further examination of certain tensile specimens after fracture revealed characteristic fracture surfaces, indicating that, although failure had occurred in the gage length, the mode of failure was by basal plane failure initiating at one edge of the gage section, propagating horizontally across the gage section. In effect, this amounts to a flexure test with loading perpendicular to the 'c' axis. This could have been easily brought about by slight misalignment or by relatively minor machine damage to the plane edges in the gage section.

Examination of edges of flexure test beams also brought out the fact that structural differences in pyrolytic graphite can produce various responses to the same grinding technique. Beams made from regenerative material showed little or no visible damage, while the surface nucleated beams nearly always had notches and chip-outs at the corners of the beams.

The lower values obtained in four-point-loading flexure tests compared to three-point-loading can thus be attributed to the fact that greater areas were included in the highly stressed portions of the former, and the probability of failure at lower stresses was proportionately increased.

Considerably more detailed attention is being given to fracture mechanisms of pyrolytic graphite and the part played by surface damage to this brittle material. A report on these considerations is currently being prepared[9].

V. CONCLUSIONS

The experimental values obtained for both surface and continuously nucleated pyrolytic graphite indicate that the material has a considerably higher and more reliable strength at room temperature (18,000 ± 2,000 psi) than has been appreciated. It is felt that this is primarily due to careful specimen preparation particularly with respect to the machining of basal plane edges. The effect of improved production techniques cannot be evaluated separately, but probably contribute to greater reliability and strength values through the availability of thicker and flatter material having fewer large nodules.

The differences between surface and continuously nucleated pyrolytic graphite with respect to response to mechanical tests occurs chiefly through the greater degree of interlocking of cones in the latter. This is brought out most clearly in torsional test results, although the same effect is apparent in flexure test results in which premature failure by delamination was characteristic of surface nucleated material. Table VI summarizes the data presented and illustrates these similarities and differences between surface nucleated and continuously nucleated pyrolytic graphite as they are currently manufactured.

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TABLE I

TENSILE STRENGTH OF PYROLYTIC GRAPHITE vs. TEMPERATURE °F

Temperature °F		CN psi	SN psi
75		17,864	17,462
		19,219	17,409
		18,296	23,677
		20,557	14,630
		13,156	17,107
	Avg.	17,818	18,191
3000		18,700	15,900
		20,600	15,800
		14,150	19,800
		20,500	16,175
	Avg.	18,488	16,919
4000		24,200	19,580
		28,600	24,800
		30,000	23,400
		27,300	20,600
	Avg.	27,525	22,095
5000		45,600	50,000
		60,500	53,400
		45,100	52,100
		69,900	46,600
	Avg.	55,275	50,525

TABLE II

TORSIONAL STRENGTH OF PYROLYTIC GRAPHITE vs. TEMPERATURE °F

Temperature °F		CN psi	SN psi
75		2333	1887
		2522	1032
		2309	1790
		2339	1484
		2887	1416
	Avg.	2578	1522
3000		2953	1476
		2182	1761
		2143	1555
		3109	447*
		2874	1631
	Avg.	2652	1374 (1606)**
4000		3070	1348
		3240	891
		3094	1740
			1570
			1795
	Avg.	3135	1469
5000		2848	915
		3214	2028
		3397	1097
		3070	1968
		3248	2090
	Avg.	3155	1620

* Did not fail in gage section.
** Average excluding low value.

TABLE III

FLEXURE TEST RESULTS
CONTINUOUSLY NUCLEATED PYROLYTIC GRAPHITE

Temperature °K		KSI			
		3 Perpendicular	3 Parallel	4 Perpendicular	4 Parallel
75		25.7	20.8	26.2	20.0
		23.4	21.8	19.9	19.1
		24.6	20.8	26.3	19.6
		23.8	22.8	23.7	22.1
		24.8	19.4	21.6	13.3
		23.6	22.6	24.4	22.3
		23.8	19.4	21.7	16.9
		25.1	21.0	21.2	20.5
		20.2	22.0	24.0	18.6
		23.8	9.7	20.6	18.7
	Avg.	23.9	20.0	23.0	19.1
3000		27.3	21.6	25.5	24.6
		28.0	23.8	25.3	20.3
		28.1	25.3	26.9	21.9
	Avg.	27.8	23.5	25.9	22.3
4000		31.0	32.3	28.7	28.4
		29.0	30.9	31.3	24.4
		33.0	33.5	33.1	31.8
	Avg.	31.0	32.2	31.0	28.2
5000		23.8NF	19.0NF	26.6NF	29.7NF

TABLE IV

FLEXURE TEST RESULTS
SURFACE NUCLEATED PYROLYTIC GRAPHITE

Temperature °K		KSI			
		3 Perpendicular	3 Parallel	4 Perpendicular	4 Parallel
75		21.6	12.7	21.3	11.2
		21.4	11.5	12.7	9.4
		20.7	20.0	18.0	10.0
		22.6	17.2	18.8	14.7
		13.4	16.1	15.7	14.3
		22.3	16.3	16.2	12.4
		17.6	18.5	20.4	6.8
		21.5	15.1	16.4	14.1
		23.9	13.5	17.2	16.4
		20.7	19.2	19.0	12.1
	Avg.	20.6	16.0	17.5	12.1
3000		20.0	14.9	20.9	14.9
		20.6	17.3	21.8	11.3
		23.0	19.9	16.3	10.9
	Avg.	21.2	17.4	19.7	12.4
4000		23.9	19.0	15.1	14.9
		24.2	22.2	18.6	16.7
		22.4	20.4	15.4	12.4
	Avg.	23.5	20.5	16.4	14.7
000		29.1	46.4NF	23.0	18.3

TABLE V. POISSON'S RATIOS AND ELASTIC MODULUS 75°F
(STRAIN GAGE DETERMINATION ON 4-POINT BEND TESTS).

Stress (KSI)	Material	Top Surface	Substrate Side	'c' Direction (Avg.)	Modulus (PSI)
10.12	CN	-0.12	-0.10	0.98	3.8×10^6
10.09	CN	-0.10	-0.09	1.07	3.4×10^6
10.15	SN	-0.16	-0.12	0.98	4.2×10^6
10.04	SN	-0.14	-0.13	1.01	4.1×10^6

TABLE VI. MECHANICAL PROPERTIES OF CURRENT PRODUCTION PYROLYTIC GRAPHITE (AVERAGE VALUES)

Test	Temp. °F		CN.	SN	
Tensile (psi)	75		17,818	18,191	
	3000		18,488	16,919	
	4000		27,525	22,095	
	5000		55,275	50,525	
Torsion (psi)	75		2,578	1,522	
	3000		2,652	1,374	
	4000		3,135	1,469	
	5000		3,248	1,620	
	Direction	Parallel	Perpendicular	Parallel	Perpendicular
Bend 3 pt(psi)	75	20,030	23,880	16,000	20,570
	3000	23,530	27,800	17,300	21,200
	4000	32,230	31,000	20,530	23,500
	5000	(no failure)	(no failure)	23,000	29,100
Bend 4 pt(psi)	75	19,110	22,960	12,140	17,470
	3000	22,270	25,900	12,370	19,670
	4000	28,200	31,033	14,670	16,370
	5000	(no failure to 29,700)	(no failure to 26,600)		(no failure to 40,400)
Elastic Modulus (10^6psi)	75		3.5	4.5	
	3000		3.4	3.5	
	4000		2.7	3.0	
	5000		1.4	2.2	
Poisson's Ratio 75°F	Substrate		-0.095	-0.13	
	Surface		-0.11	-0.15	
	'c' direction		1.02	0.99	
Coeff. of Expansion (in/in/°F)					
'a' 2500-4500°F			2.1×10^{-6}	2.0×10^{-6}	
'c' RT - 4500°F			14.2×10^{-6}	15.0×10^{-6}	

(a) Surface Nucleated Pyrolytic Graphite

(b) Continuously Nucleated Pyrolytic Graphite

Figure 1. Microstructure of Surface Nucleated and Continuously Nucleated Pyrolytic Graphite

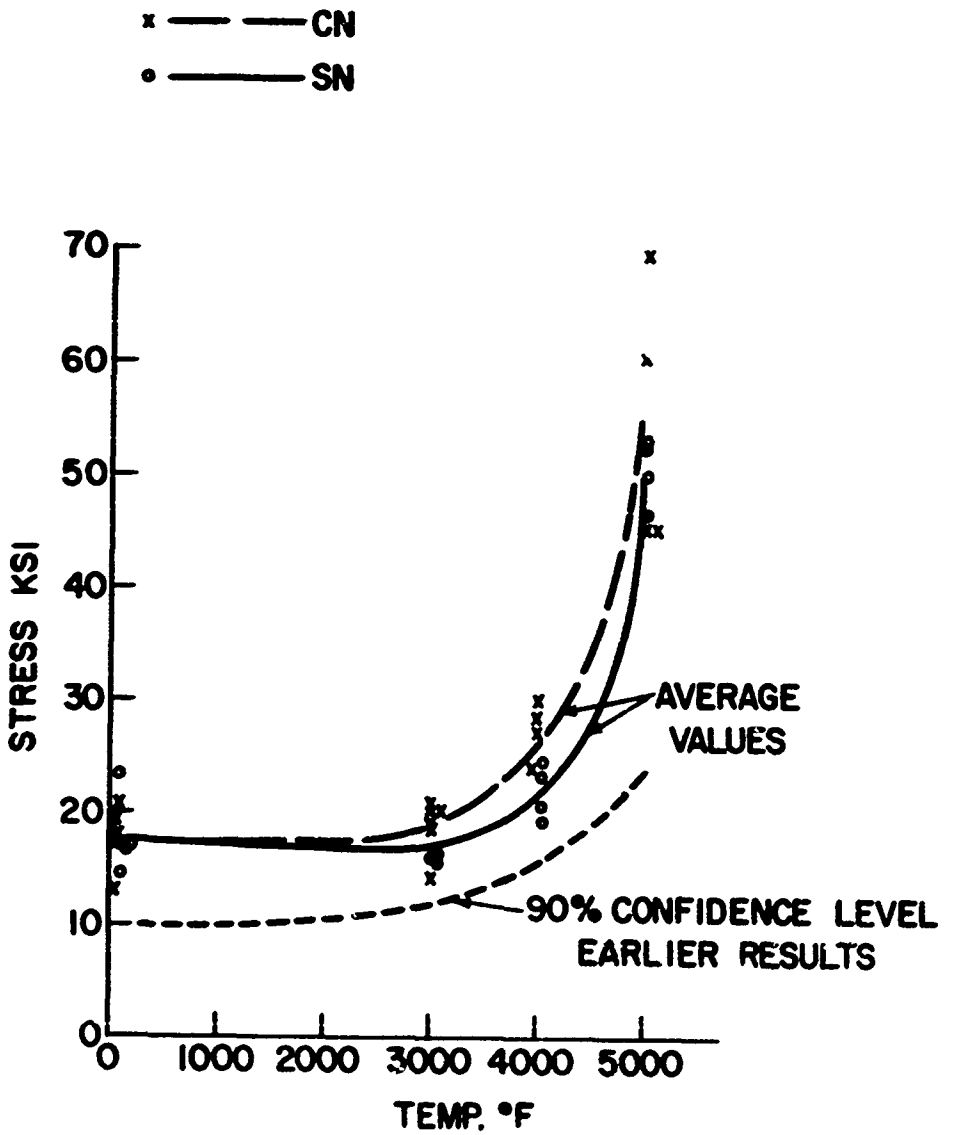

Figure 2. Tensile Strength of Pyrolytic Graphite vs. Temperature °F.

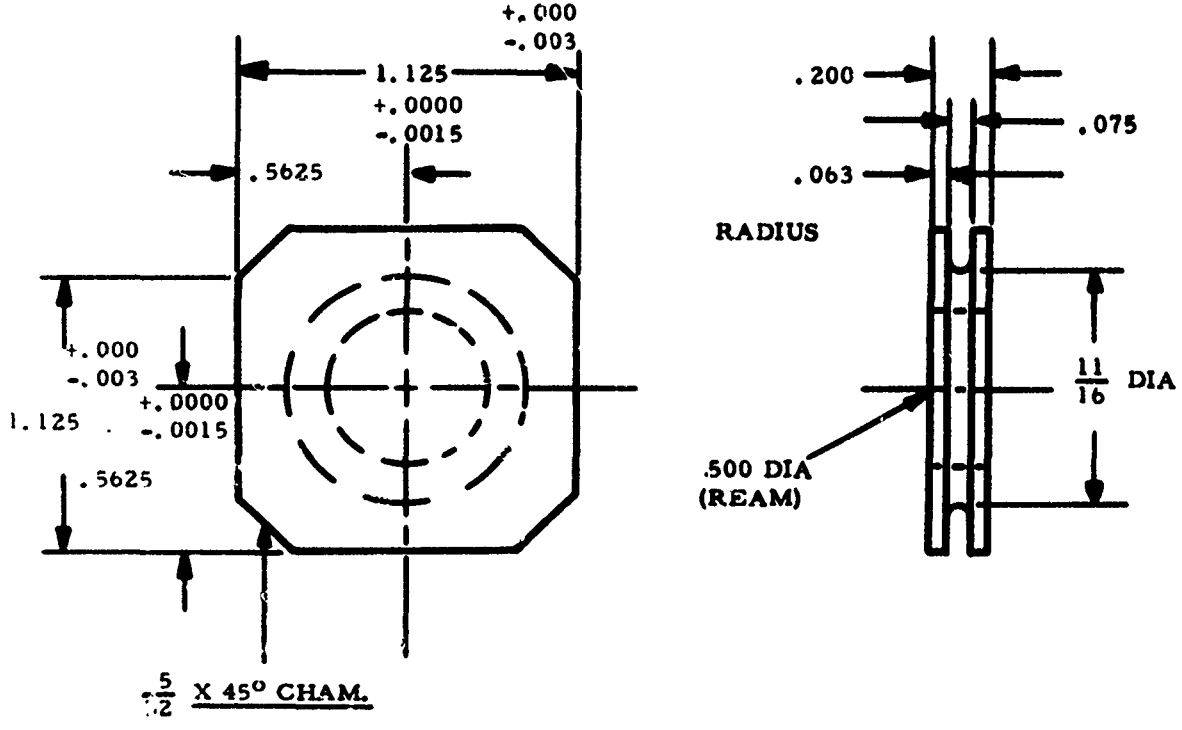

Figure 3. Torsion Test Specimen

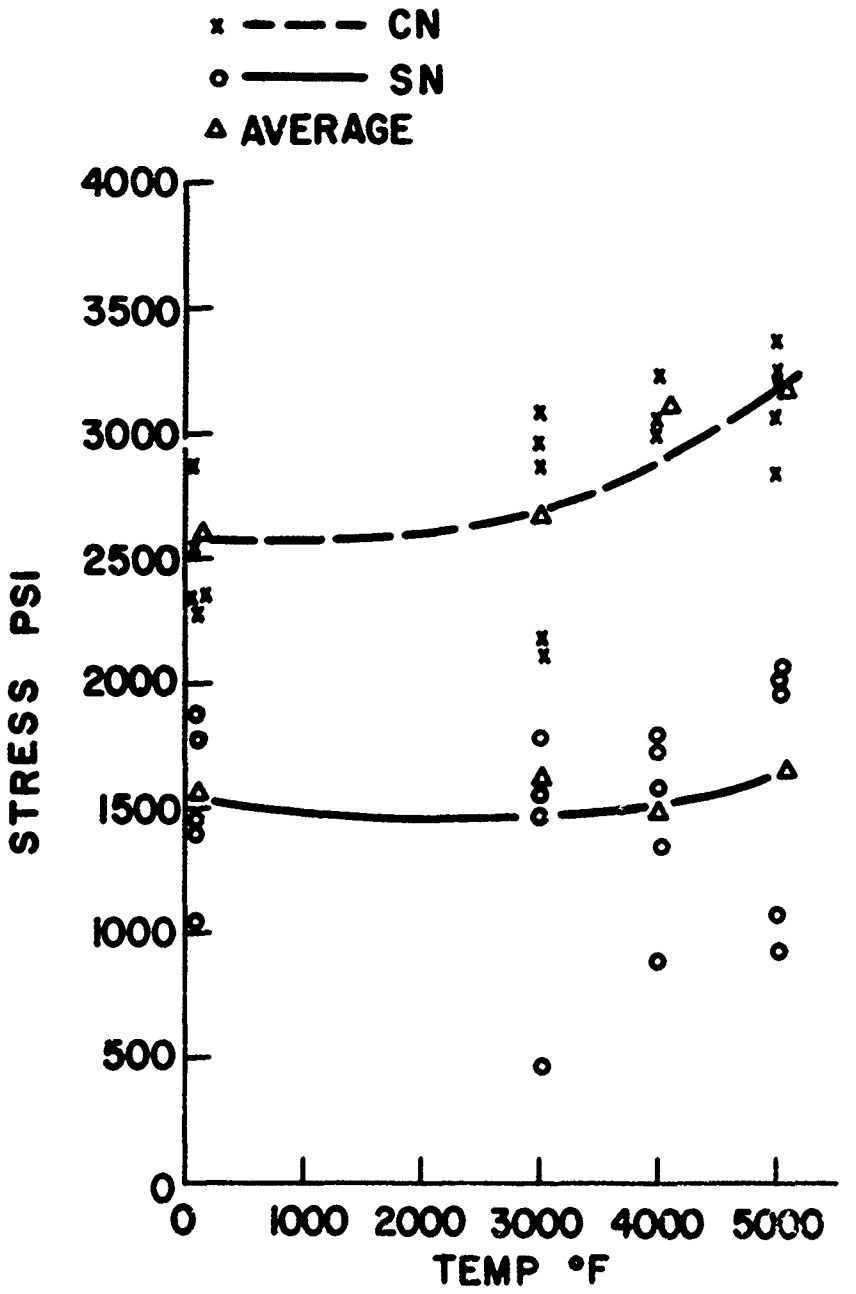

Figure 4. Torsional Strength of Pyrolytic Graphite vs Temperature.

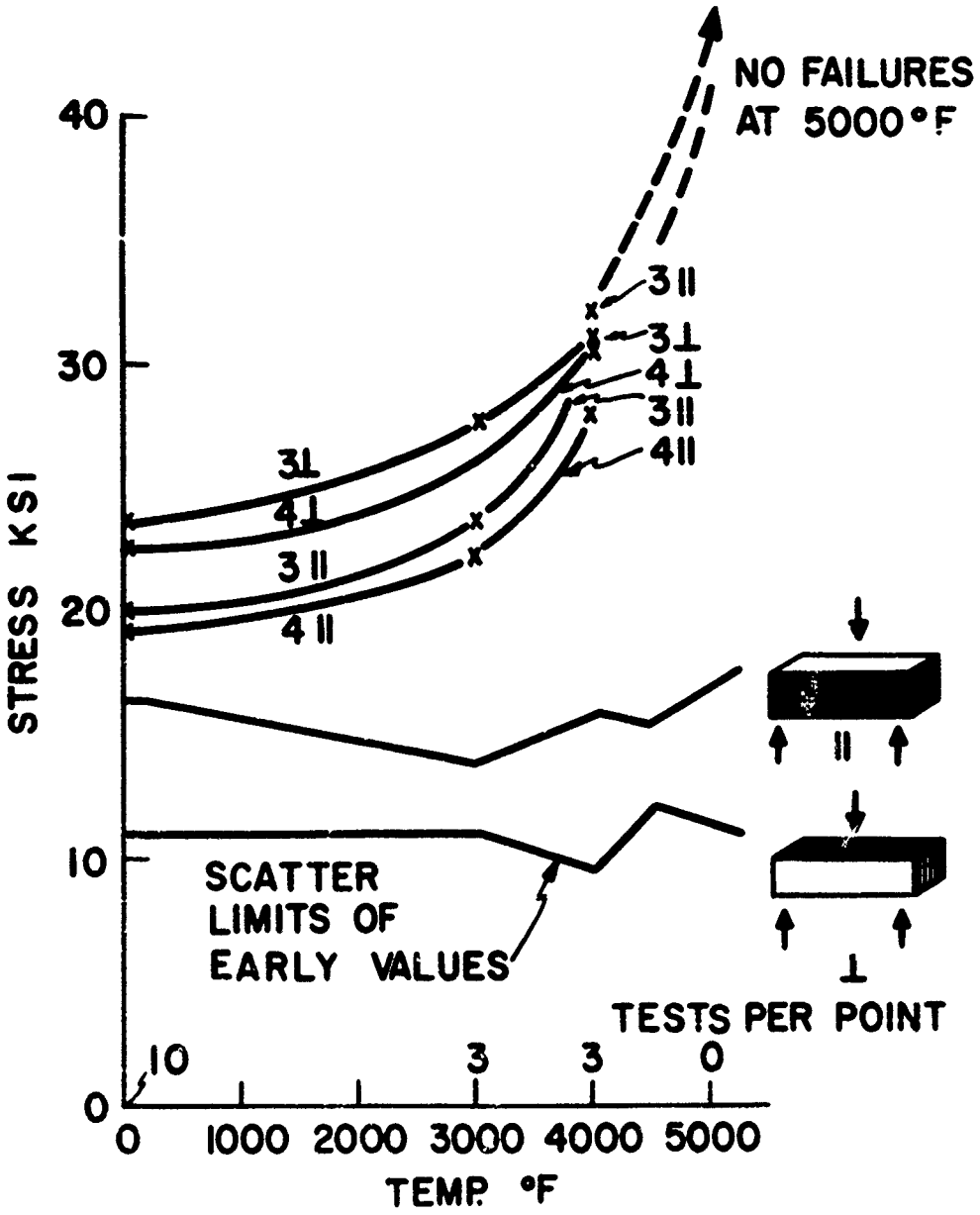

Figure 5. Average Flexure Strength of Pyrolytic Graphite (Continuously Nucleated) vs Temperature °F. 3- and 4- Loading.

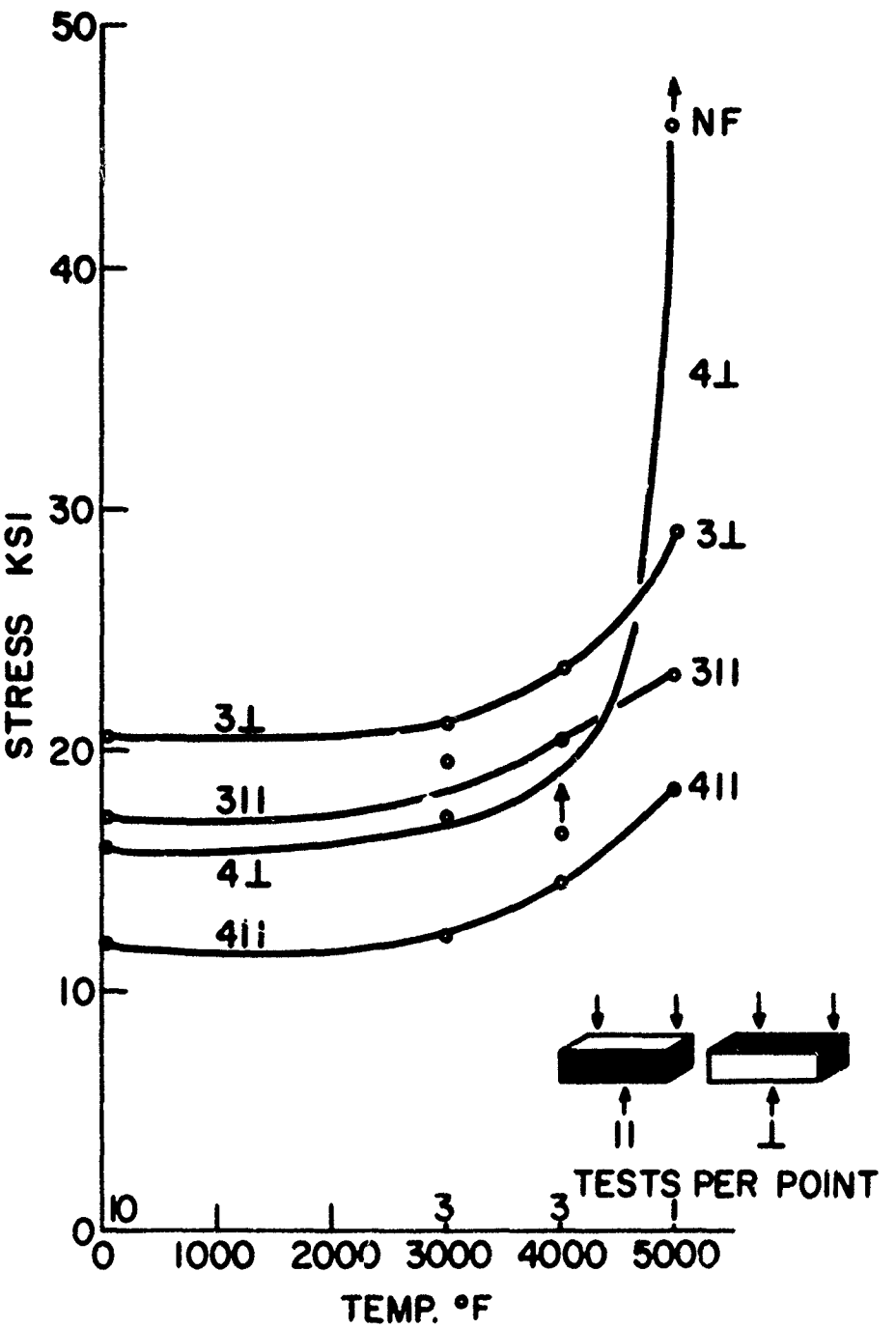

Figure 6. Average Flexure Strength of Pyrolytic Graphite (Surface Nucleated) vs. Temperature °F.

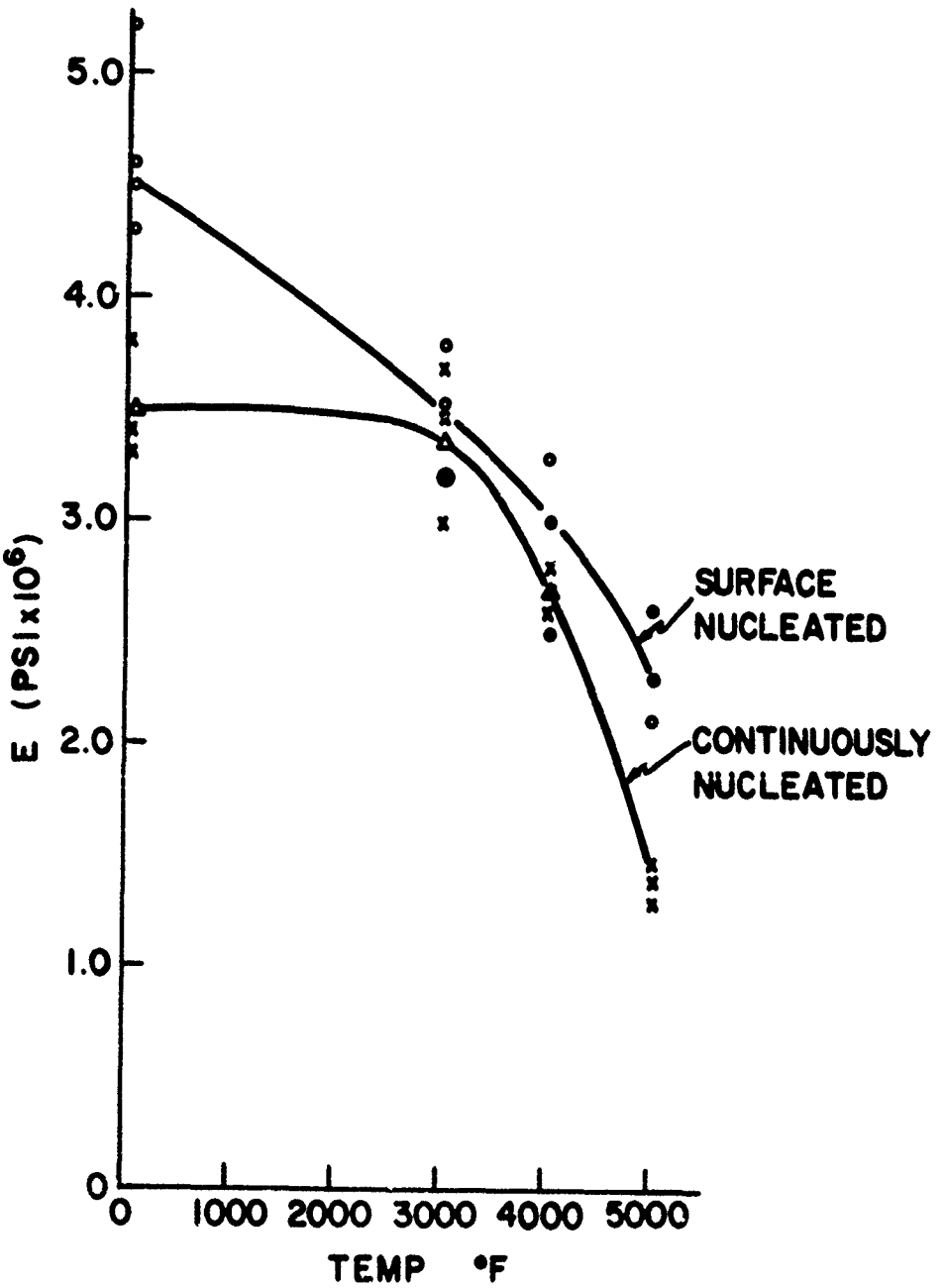

Figure 7. Elastic Modulus Pyrolytic Graphite 'a' Direction vs. Temperature

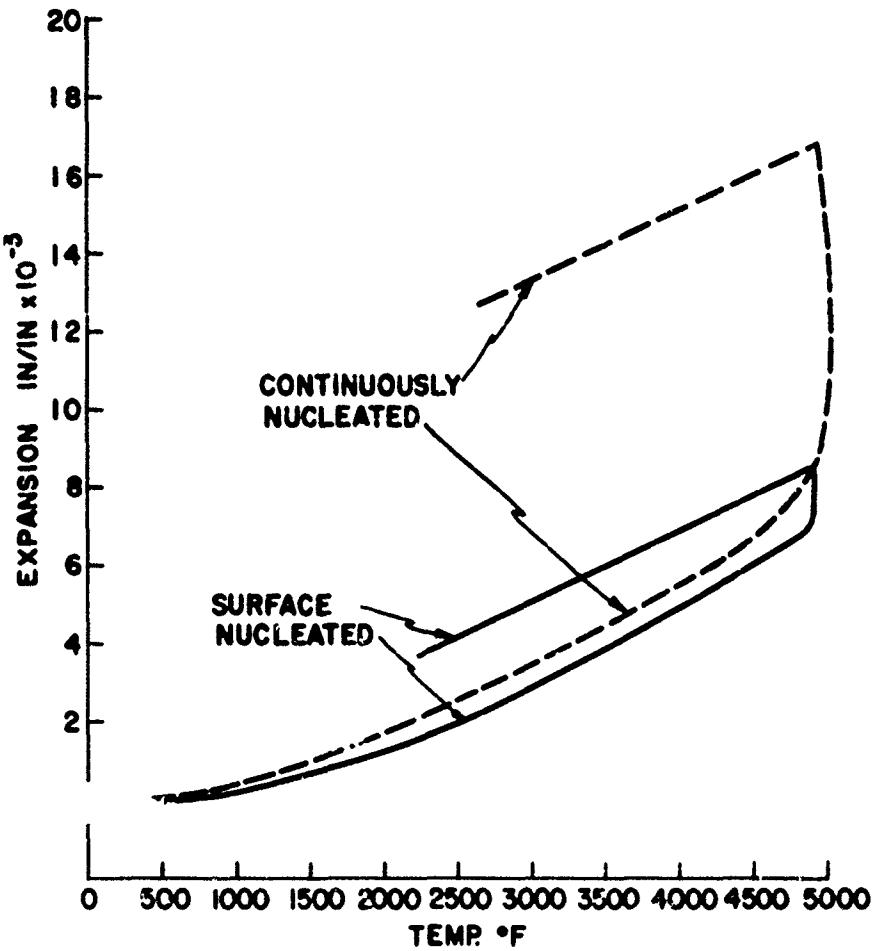

Figure 8. Thermal Expansion Pyrolytic Graphite 'a' Direction vs. Temperature.

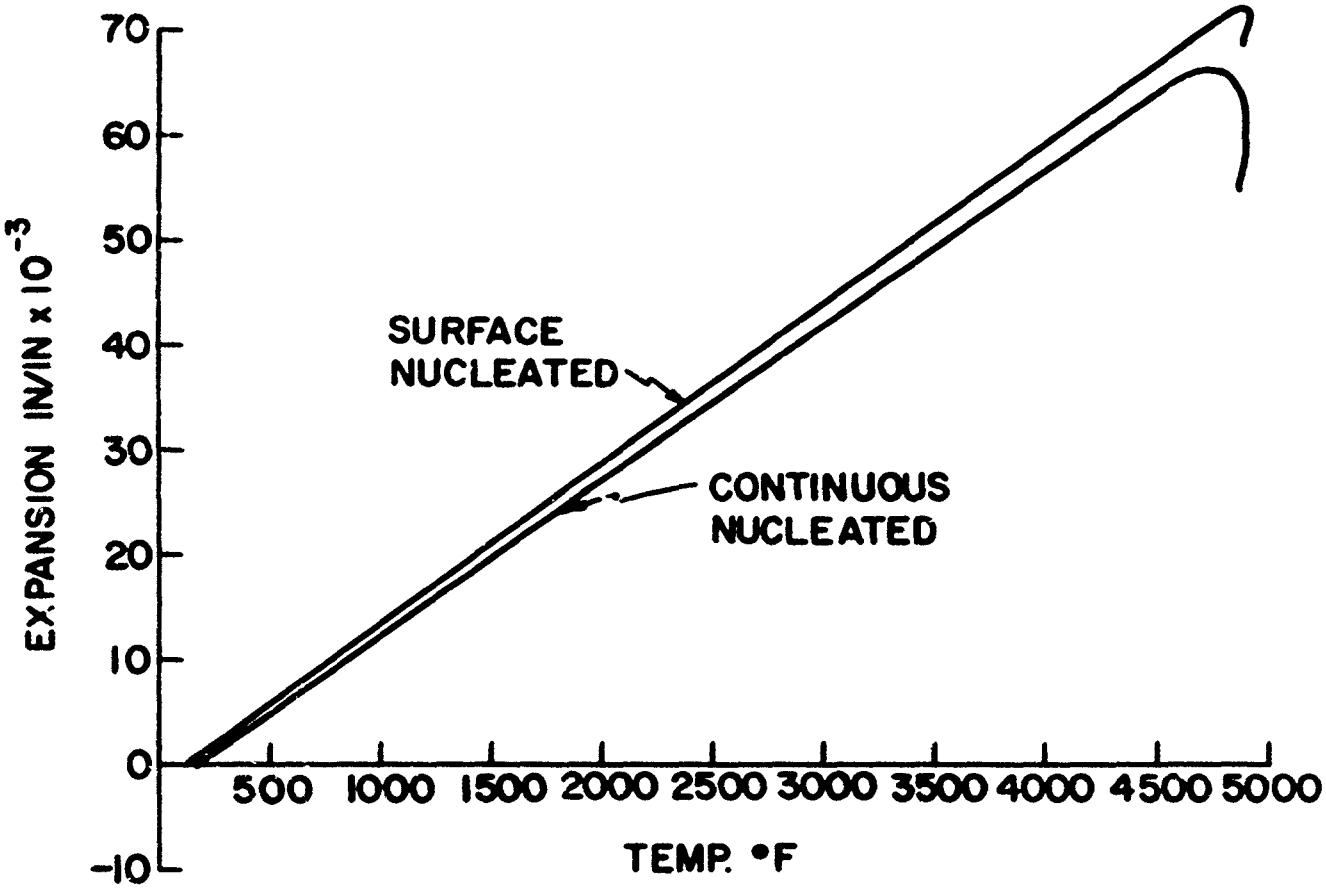

Figure 9. Thermal Expansion Pyrolytic Graphite 'c' Direction vs. Temperature °F.

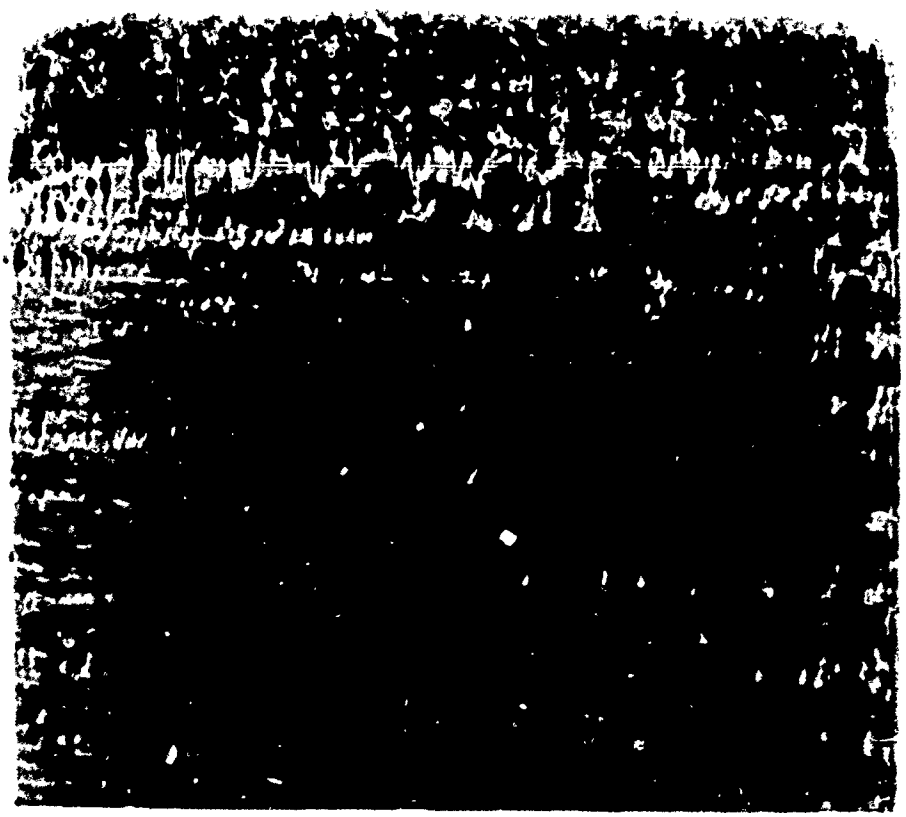

Figure 10. Damaged Region Around Grip-Hole in Tensile Specimens (Flame Polished) (from Ref. 11)

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SUMMARY A series of mechanical strength and thermal expansion tests has been carried out from room temperature to 5000°F on the two microstructural types of current production pyrolytic graphite. Results indicate that with appropriate precautions in selecting test material, machining and testing specimens, current production pyrolytic graphites yield higher, more reliable mechanical property values. The contribution of process improvements to this increase cannot be specifically separated although it may show up principally in allowing more of a given batch of material to be selected. The present series of tests shows pyrolytic graphite to have an ultimate 'a' direction tensile strength of 18,000 ± 2,000 psi at room temperature rather than previously found values of 11,000 ± 5,000 psi. The largest difference between surface nucleated and continuously nucleated material was obtained in torsional tests parallel to the planes; values of 1555 ± 60 and 2880 ± 255 psi were obtained respectively throughout the temperature range to 5000°F.

KEY WORDS
Pyrolytic Graphite, Mechanical Properties

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